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CENTRAL INTELLIGENCE AGENCY

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INTRODUCTION

1. In early 1946 at Sinop Institute, apparatus was developed in the laboratory of /Dr. Hans/ Bartel and /Ing. Wladimir/ von Maydell of Peter Adolf Thiessen's department in order to test barrier by a pressure equalization method. The diaphragm was placed in a holder which formed the connection, between two glass bulbs. Manometers were provided to measure the pressure in each bulb and the drop in pressure across the barrier. no details as to the operation of this experiment, but certain that it did not lead to any satisfactory result.
2. Somewhat later, the Bartel and von Maydell section developed another apparatus for measuring the permeability of barrier by a continuous flow method. In this equipment, the barrier was

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placed in a holder which was connected on one side to a needle valve which admitted air from the room to the system; a needle valve connected to the other side of the holder was, in turn, connected to a fore vacuum pump. An absolute mercury manometer was provided to measure the pressure on the upstream side of the barrier; an oil differential manometer served to measure the pressure drop across the barrier. The manometers which were provided for measuring the absolute pressure and the pressure drop across the porous diaphragm were longer than those furnished with the so-called standard measuring apparatus used later. The cross-sectional area of the manometer's tubing and the length of the connecting rubber hose were smaller. There was a "considerable discrepancy" between the values which were measured at Sinop with this early equipment and those which were measured in Moscow.

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3. Sometime later, at an unrecalled date (early in 1947), a standard measuring apparatus was received in Sinop from a Soviet laboratory believed to have been located in Moscow. This apparatus was accompanied by etalon barriers, so that it was possible to check the constant of the apparatus without difficulty. The procedure for measurement of γ (gamma) and $\Delta\gamma$ (delta gamma over gamma) and their definitions were specified at the same time, but source does not recall seeing any written instructions.
4. The permeability of a barrier was characterized by a quantity called gamma. This was recalled to be a dimensionless constant which is proportional to the flow of air through a unit area of the barrier for a given pressure drop across the barrier. In practice, the gamma value was determined from the experimentally determined pressure drop across the barrier for a known constant flow of air through the barrier. Thus the gamma value is equal to the apparatus constant divided by the pressure difference across the diaphragm.³
5. A second barrier constant which was determined was $\Delta\gamma$ (delta gamma over gamma). This quantity is a measure of the change in permeability (gamma value) over a prescribed pressure range.

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3. A quantitative discussion of the theory of flow through a barrier and the definition of gamma are given on page 16 of this report.

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This term is a measure of the ratio of Knudsen flow to Poiseuille flow, and is related to the average radius of the pores in the diaphragm.⁴ This quantity may be defined as

$$\frac{\Delta \gamma}{\gamma} = \frac{\gamma_2 - \gamma_1}{\gamma_1}$$

Because γ is inversely proportional to the pressure drop across the barrier, this reduces to

$$\frac{\Delta \gamma}{\gamma} = \frac{\Delta p_1 - \Delta p_2}{\Delta p_1}$$

6. This constant is determined on a slide rule by simply dividing the difference between the pressure drop (measured at the two values of the fore pressure) by the value of the pressure drop which was recorded for the lower pressure.
7. [redacted] the measurement of the gamma value was specified at an upstream pressure of 15 mm of mercury. [redacted] however, [redacted] an early period this constant was measured at a higher pressure, perhaps 35 mm of mercury, because the lower pressure could not be attained with the small fore pump then in use. [redacted]
[redacted] the end of the column was on a "half line" on the scale which appeared at every 5 millimeter interval. ("Whole lines" occurred at the 10 millimeter intervals). On this basis, [redacted] the higher pressure was either 55 mm, 65 mm, or 75 mm of mercury. [redacted] the 65 mm value the most probable.
8. The measuring apparatus supplied by Moscow was modified by the Bartel-von Maydell section so as to adapt the equipment to measuring tubular barrier. This equipment is described in detail on pages 5 to 10 of this report.
9. As an adjunct to the barrier production process being developed by the Technological Section of the Thiessen Department, a "gamma forceps" (Gamma Zange) was developed. This was a small

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4. The relation of delta gamma to the pore radius is given on pages 16 and 17.

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unit which could be clamped on a sheet of barrier to measure the permeability of a small area. In practice, the gamma values (and delta gamma over gamma values) were measured at two or three places on the sheet of barrier before it was rolled into a tube. The differences between the three values were a measure of the homogeneity of the barrier. The difference between the gamma value of the flat diaphragm and that of the finished tube was a measure of the tightness of the welded seam.

10. A second Soviet apparatus was encountered [redacted] sometime in 1949. It was used in production testing. This apparatus was identical to the apparatus which was developed in Sinop except for the measuring head, which was vertical rather than horizontal. [redacted] these units in the factory at Elektrostal. Later, apparatus of this type was received in Sinop.

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DESCRIPTION OF STANDARD MEASURING APPARATUS

11. A schematic diagram of the arrangement of the apparatus is given in Figure 1 on page 6. The following items are noted on this sketch:

Point 1 Manometry

A detailed sketch of the manometry is given in Figure 2 on page 7. This is identical to the arrangement provided with the original equipment received from Moscow. It was taken over without modification, and was also used with the "gamma forceps" and the so-called vertical measuring apparatus. It should be noted that the manometers in this apparatus are relatively short.⁷ Care was taken to make the cross section of the tubing large, so that the flow resistance in the apparatus was as small as possible. In this case, no measurable pressure drop was observed on the oil manometer when the machine was operated

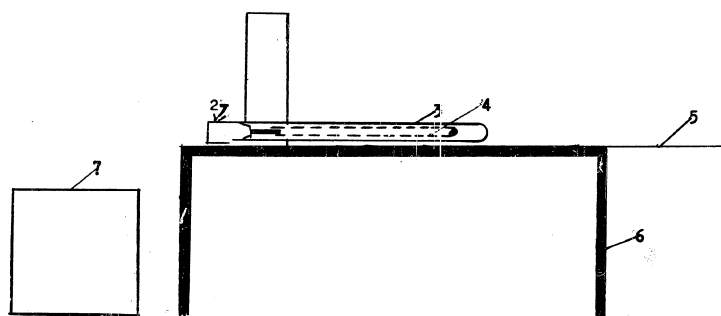
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7. The restriction in the range of gamma values over which the apparatus could be used is discussed on pages 13 and 18-21.

/Text continued on page 8/

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Scale 1:1

SCHEMATIC ARRANGEMENT

Fig. 1

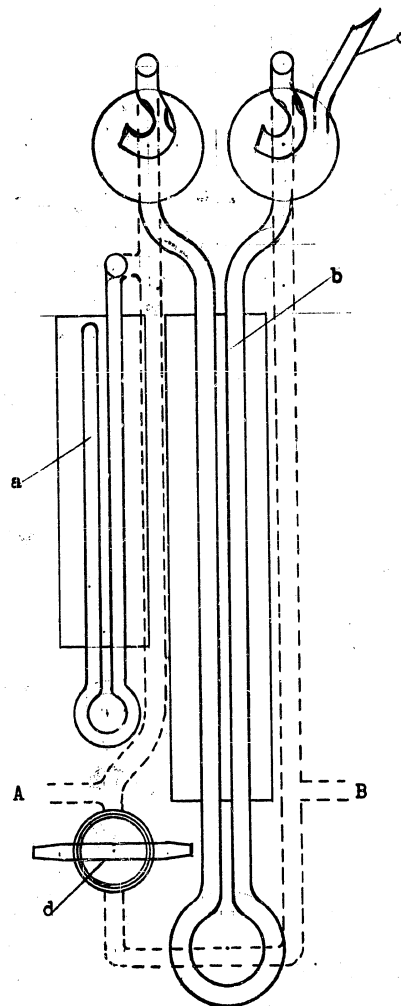
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FIGURE 2



Scale 1:2

STANDARD MEASURING APPARATUS

DETAIL OF MANOMETER

ARRANGEMENT

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without a barrier. In the earlier equipment, a correction to the reading was necessary to take account of this effect.

One experimental apparatus in Bartel's laboratory was fitted with a set of long manometers. The purpose of this apparatus was to measure gamma at higher pressures in order to extrapolate the gamma-zero value (value of gamma at zero pressure). the group was also studying whether the values obtained at higher pressures had any real meaning or whether, in this region, only Poiseuille flow existed rather than Knudsen flow.

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Point 2

Measuring Head

A detailed sketch of the measuring head is given in Figure 3 on page 9. Holes A and B were connected, by means of thick-walled rubber tubing of large diameter, to corresponding points on the manometry. See Figure 2 on page 7. C is the conical sleeve on which the tubular barriers were fitted. E is a small brass disk in which one, two, or three small holes were punched. These holes served as critical orifices to meter the flow of air into the instrument. The holes were punched in a very thin section at the center of the disk.⁹ the holes were under one-tenth of a millimeter in diameter. the disks were manufactured by Wolfgang Friedrich Srocke.¹⁰ the volume of air per unit time which passed through the orifice was measured with a gas meter.

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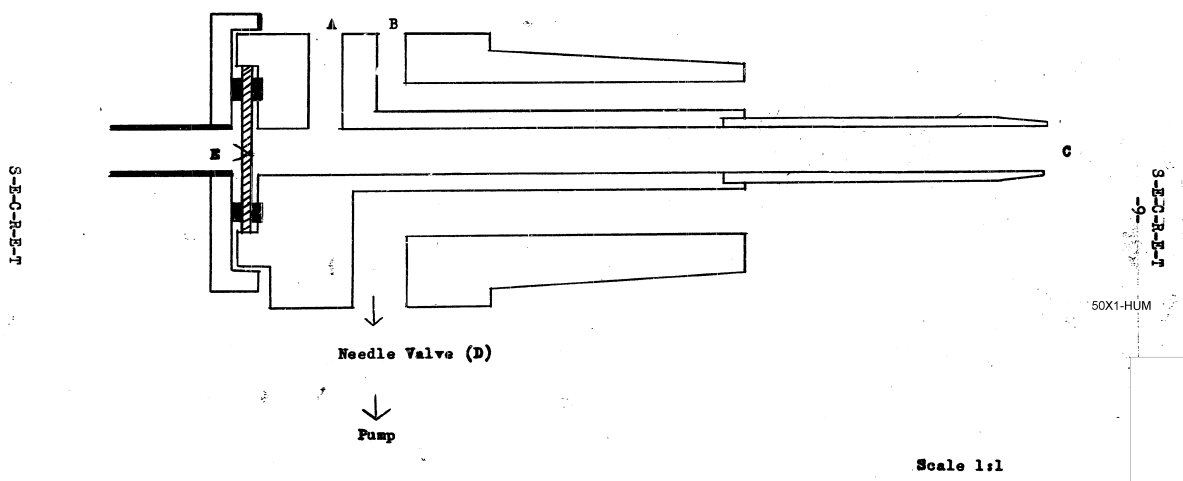
9. [redacted] mechanic made brass plates which were very thin in the middle. This mechanic then punched holes in this plate Probably with a needle And these plates were placed in the measuring apparatus and then tested for their volume per unit time. Now, such plates were selected from the batch which gave an oil deflection corresponding to the expected value of gamma" (for the standard manometry).
10. Srocke was one of the best mechanics at Sinop. Shortly after [redacted] returned to the DDR, [redacted] employed Srocke in [redacted] Institute of Catalysis Research in Berlin-Liebenwalde.

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DETAIL OF HEAD OF STANDARD MEASURING APPARATUS

FIGURE 3

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Point 3 Glass Outer Tube

This tube was provided with a flared conical opening which slipped over the measuring head.

Point 4 Barrier

The tubular barrier was slipped on the corresponding sleeve on the measuring head. A small soft aluminum closure was provided for the other end of the tube.

Point 5 Rest for Glass Tube

A small extension shelf was provided for holding the glass outer tube when it was not in use.

Point 6 Bench

The apparatus was mounted on a small wooden table.

Point 7 Vacuum Pump

A medium size Kinney-type pump was provided to evacuate the system during test. During the early stages in the development of this test equipment, the vacuum pumps then available would not permit measurements of gamma at pressures lower than perhaps 35 mm.

TEST PROCEDURE

12. The following procedure was employed in testing a diaphragm with the measuring apparatus:
 - a. Diaphragm was mounted.
 - b. The glass receptacle was put over diaphragm.
 - c. The valve across the oil manometer (indicated as (d) in Figure 2 on page 7) was opened.
 - d. The pump was started.
 - e. Fifteen millimeter pressure was set for the absolute mercury manometer by adjustment of the needle valve.
 - f. Valve across oil manometer was closed.
 - g. The pressure difference across the oil manometer was read, and the gamma value at 15 mm pressure was calculated.
 - h. The valve across the oil manometer was opened.

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- i. Sixty-five millimeter pressure was set for the absolute mercury manometer by means of the needle valve.
- j. The value across the differential oil manometer was closed.
- k. The pressure difference across the oil manometer was read, and delta gamma over gamma for the range 15 mm to 65 mm Hg was computed.

PHYSICAL PROPERTIES OF THE STANDARD THIessen BARRIER 11

13. The basis of the production was 10,000 mesh per square centimeter of nickel wire. Other screen with 5,000 to 20,000 mesh was tried, but 5,000 mesh was found to be too coarse and 20,000 was too expensive. In 1947, 10,000 mesh was adopted. The 10,000 mesh screen came from an unidentified factory in the DDR through the First Chief Directorate.
14. The screen arrived in rolls of about 100 cm width. The wire drawing lubricant was removed by [sodium-hydroxide] lye, and the mesh was then etched in acid to give a 20 percent decrease in weight.
15. The screen was rolled, which produced a one-percent extension in the direction of rolling and some reduction in thickness, together with a related variation in the volume of the holes. The thickness of the rolled screen and the finished diaphragm was measured on a polished metal plate with a dial gauge.
16. The wire diameter was 0.05 mm with a small (unknown) tolerance. For a surface area of 250 cm² (12) and an assumed 200 cm of wire per cm², the weight of the screen was

$$W = \rho \cdot A \cdot 200 \cdot \frac{d^2 \pi}{4} = \frac{8.9 \times 250 \times 200 \times (0.005)^2 \pi}{4}$$

$$W = 8.75 \text{ gm.}$$

11. This summary of the properties of the barrier was made to be used as a basis of calculations in paragraphs 26 to 28. A further discussion of this summary is found on page 21.

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12. This is the area of wire mesh required for one Thiessen tubular barrier 1.5 cm in diameter and 50 cm long including the seam for welding.

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17. Because the wire was woven, [redacted] 224 cm of wire would be required for a square cm of 10,000 mesh screen (an increase of 12 percent). The corresponding weight for 250 cm² was

$$W = 9.8 \text{ gm.}$$

The 20 percent loss caused by etching gave

$$W = 7.85 \text{ gm.}$$

18. The volume occupied by this wire was therefore

$$V = \frac{W}{\rho} = 0.88 \text{ cm}^3$$

[redacted] the thickness of the wire mesh after rolling to be 0.085 mm, and calculated the total volume to be 2.11 cm³. The volume available to be filled with sintered nickel powder was estimated to be

$$2.11 - 0.88 = 1.23 \text{ cm}^3$$

19. [redacted] the density of the sinter material was approximately 50 percent that of solid nickel. [redacted] this is the density obtained for a nickel powder sintered at a temperature which is two-thirds its melting temperature.

20. The weight of the sintered nickel powder was

$$1.23 \times 4.45 = 5.5 \text{ gm.}$$

The total weight of the barrier was estimated to be

$$5.5 + 7.85 = 13.35 \text{ gm.}$$

This compared well with a value of 13 grams which was recalled for this weight.

21. The weight of a solid nickel tube of the same dimensions would be

$$8.9 \times 2.11 = 18.9 \text{ grams.}$$

Therefore, 70 percent of the barrier is nickel, and the pore volume is accordingly 30 percent of the total volume. The porosity ξ is by definition

$$\xi = \frac{1}{0.3} = 3.3$$

THEORY OF THE FLOW OF GAS THROUGH A BARRIER

22. Prior to the reconstruction of the theory of the flow of gas through a diaphragm, source recalled the following:

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- a. The details of the construction and operation of the apparatus for determination of gamma and delta gamma over gamma. [redacted] the flow of gas through the diaphragm was proportional to (a) the pressure drop across the diaphragm, (b) a constant, and (c) the permeability of the diaphragm (its gamma value).

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- b. The gamma value of a Thiessen tubular diaphragm was recalled with certainty to be about 1×10^{-3} . The corresponding pressure drop in the apparatus was recalled to be about 100 mm of "bone oil". The "apparatus constant K " defined from the equation

$$\gamma = \frac{K}{\Delta p_{oil}} \times 10^{-3}$$

was recalled to be between 100 and 200. This was recognized to be dependent on the mass flow through the critical orifice, the area of the diaphragm, the barometric pressure, the ambient temperature, the mean free velocity of the molecules, the humidity, and the gas viscosity.

- c. The flow of dry nitrogen through a tubular diaphragm in the measuring apparatus which corresponds to a pressure drop across the diaphragm of 100 millimeters of oil was recalled to be 120 cm³/sec.
- d. The gamma value was known to be a coefficient characterizing the intrinsic properties of the barrier including the average pore length, the average pore radius, and the number of pores per square centimeter. The porosity of the diaphragm was introduced into this reconstruction at a later point.
- e. The equation for the molecular (or Knudsen) flow of gas through a capillary of length l and radius r which is given on page 1499 of J. Dans and E. Lax Taschenbuch fur Physiker und Chemiker was used as the basis for this theory, when it was outlined to source by Bartel.

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23. The following outline of the theory of flow through a barrier was developed only after a number of blunders. See footnote 15 for an example.

24. From D'Ans and Lax Taschenbuch für Chemiker und Physiker, page 1499, the mean velocity \bar{w} in a pore of radius r and length l , satisfying the condition for pure Knudsen flow (viz., λ (mean free path) $> r$) is

$$\bar{w} = \frac{1}{\sqrt{\rho_1}} \cdot \frac{4}{3} \cdot \frac{r}{l} \sqrt{\frac{8}{\pi}} \frac{p_1 - p_2}{p_1 + p_2}$$

\bar{w} = velocity of flow, (cm sec⁻¹)

ρ_1 = gas density at cm² dyne cm⁻² pressure, (gm cm⁻³)

r = pore radius, (cm)

l = pore length, (cm)

p_1 = upstream pressure, (mm Hg)

p_2 = downstream pressure, (mm Hg)

Defining the mass flow through a diaphragm made up of identical pores

$$\frac{M}{t} = A N \rho a \bar{w}$$

A = area of barrier, (cm²)

N = number of pores per cm², (cm⁻²) - assumed identical

ρ = gas density at mean pressure $\frac{p_1 + p_2}{2}$, (gm cm⁻³)

$a = \pi r^2$ = cross section area of one pore, (cm²)

On substitution

$$\frac{M}{t} = \frac{1}{p_1 + p_2} \pi \rho \frac{1}{\sqrt{\rho_1}} \frac{4}{3} \sqrt{\frac{8}{\pi}} A \frac{r^3}{l} N (p_1 - p_2)$$

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25. At this point in the analysis, source tentatively identified

$$\gamma' = \frac{r^3}{2} N$$

as gamma. However, the term was not familiar in this form. A consideration of the meaning of N and its determination by measurements which could be made on the diaphragm led source to recall the porosity.¹⁴

$$\epsilon = \frac{\text{Total volume}}{\text{Pore volume}}$$

$$\epsilon = \frac{A l}{A N \pi r^2 l}$$

and

$$N = \frac{1}{\epsilon \pi r^2}$$

On substitution of this term in the $\frac{M}{t}$ equation in paragraph 24, then:

$$\frac{M}{t} = \frac{1}{p_1 + p_2} \rho \frac{1}{\sqrt{p_1}} \frac{4}{3} \sqrt{\frac{8}{\pi}} A \frac{r}{2\epsilon} (p_1 - p_2)$$

and source identified

$$\gamma = \frac{r}{2\epsilon}$$

as the definition of gamma.

Thus,

$$\frac{M}{t} = \rho \frac{1}{\sqrt{p_1}} \frac{4}{3} \sqrt{\frac{8}{\pi}} A \frac{p_1 - p_2}{p_1 + p_2} \cdot \gamma$$

$$\rho = \rho_{760} \left(\frac{p_1 + p_2}{2 \cdot 760} \right)$$

ρ_{760} = density of gas at pressure of one atmosphere

$$\text{Thus, } \frac{M}{t} = A \frac{\rho_{760}}{760} \frac{1}{\sqrt{p_1}} \frac{2}{3} \sqrt{\frac{8}{\pi}} \cdot \Delta p \gamma$$

The volume flow V/t ($\text{cm}^3 \text{sec}^{-1}$), into the pores of a diaphragm may be expressed as

$$\frac{V}{t} = \frac{M}{\rho_{760}} = \frac{A}{760} \frac{1}{\sqrt{p_1}} \frac{2}{3} \sqrt{\frac{8}{\pi}} \Delta p \cdot \gamma$$

14. [redacted] the definition of ϵ in the article of Pecukkas and Gage [redacted] for powder size determination.

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APPLICATION OF THEORY TO THIESSEN STANDARD BARRIER

Flow through Diaphragm under Test Conditions

26. [] $A = 250 \text{ cm}^2$, $\gamma = 1 \times 10^{-3}$,
 $P_1 - P_2$ at 100 mm of oil = 6.5 mm mercury.

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$$\frac{M}{t} = \frac{0.0012 \times 250 \times 1.59 \times 6.5 \times 10^{-3} \times 2}{760 \times 3.5 \times 10^{-2} \times 3} = 7.81 \times 10^{-2} \text{ gm sec}^{-1}$$

and the calculated value of the volume flow would be

$$V = \frac{M}{\rho_{760}} = \frac{7.81 \times 10^{-2}}{0.0012} = 65 \text{ cm}^3 \text{ sec}^{-1} \text{ of air at 760 mm } 20^\circ\text{C}.$$

This does not agree with the value of $120 \text{ cm}^3 \text{ sec}^{-1}$ []

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27. Calculation of Pore Size

[] a Thiessen diaphragm would have $\gamma = 1 \times 10^{-3}$,
 $\eta = 0.85 \times 10^{-2} \text{ cm}$, and $\xi = 3.3$. Therefore, the "average" pore

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15. [] Note:

[] two mistakes in arithmetic in []
this analysis. [] density of the gas passing
through the diaphragm as $0.0012 \text{ gm cm}^{-3}$, i.e., at 760 mm Hg,
when it should have been

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$$\frac{0.0012 \times (P_1 + P_2)}{760 \times 2}, (P_1 \text{ and } P_2 \text{ in mm Hg})$$

$$\sqrt{\rho_1} = \sqrt{0.0012 \times 10^{-6}} = 3.5 \times 10^{-4}$$

instead of 3.5×10^{-5}

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[] quantity [] turned out to be
nearly equal to the value of $120 \text{ cm}^3 \text{ sec}^{-1}$ []

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radius

$$r = \gamma \epsilon = (1 \times 10^{-3}) (0.85 \times 10^{-2}) (3.3) \\ = 0.3 \times 10^{-4} \text{ cm} = 0.3 \text{ micron}$$

and a diameter of 0.6 micron. 16 This value agrees with the recollection of source that the pore size achieved with the Thiessen barrier was under one micron.

Calculation of Apparatus Constant

28. Solving the equation

$$V = \frac{A}{760} \frac{1}{\sqrt{p_1}} \frac{2}{3} \sqrt{\frac{8}{\pi}} \Delta p \gamma$$

for gamma, one obtains

$$\gamma = \frac{V \times 760 \sqrt{p_1} \times 3}{2 \times A \times 1.59} \cdot \frac{1}{\Delta p} \quad [\Delta p \text{ in mm Hg}]$$

Substituting

$$V = 120 \text{ cm}^3 \text{ sec}^{-1}$$

$$A = 250 \text{ cm}^2$$

$$\sqrt{p_1} = 3.5 \times 10^{-5}$$

one obtains the expression

$$\gamma = \frac{120 \times 760 \times 3.5 \times 10^{-5} \times 3}{250 \times 2 \times 1.59} \cdot \frac{1}{\Delta p}$$

which reduces to

$$\gamma = 1.20 \times 10^{-2} \frac{1}{\Delta p}$$

for Δp expressed in millimeters of mercury.

If Δp is expressed in millimeters of oil [] the oil used to be bone oil which has a density of about 0.85, then

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$$\gamma = 0.19 \frac{1}{\Delta p \cdot \text{oil}} \quad \text{or} \quad \gamma = \frac{190}{\Delta p \cdot \text{oil}} \times 10^{-3}$$

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16. [] estimation of pore radius from this equation is described in paragraphs 38 to 62. In these a value of $\epsilon = 2.0$ was used.

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the apparatus constant was between 100 and 200.

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QUANTITATIVE SUMMARY OF INTELLIGENCE RELATING TO APPARATUS CONSTANTS

29. An endeavor was made to get [] maximum and minimum figures to give most likely values for apparatus flow rate. The variations in upper test pressure were from 55 to 75 mm Hg, but these did not make any difference to the flow rate in terms of cc standard air/sec, as they cancelled out in the equation of flow. In any case, the lower test pressure remained constant at 15 mm. The discrepancy, therefore, lay in γ and Δp , or in the theoretical model chosen.

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30. The range of specification γ was $0.8 - 1.2 \times 10^{-3}$ at first, and then later $1.0 - 1.4 \times 10^{-3}$. For constant $\Delta p = 100$ mm oil this would give a variation from

$$0.8 \times 65 \text{ to } 1.4 \times 65 \\ = 52 \text{ to } 91 \text{ cc/sec}$$

31. [] the specification range of γ could not explain the discrepancy, inasmuch as to give 120 cc/sec at the stated Δp of 100 mm oil, the value of γ would have to be $\frac{120}{65} \times 10^{-3} = 1.8 \times 10^{-3}$. [] the only figures affecting the calculated test γ were air flow rate, pressure drop across the barrier, barrier area, and apparatus constant K (which included barrier area and flow rate).

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17. [] the relation between this theory and the tests is described in paragraphs 29 to 37.

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watch value of 120 cc/sec was true of the corrosion tests, where γ was in the range 1.0, 1.2, 1.4, 1.6, 1.8×10^{-3} . Tubes were given further sintering to get higher γ values. Only one barrier tube was tested at a time. The oil manometer was never below 40 mm oil.

32. [] the highest and lowest Δp associated with $\gamma = 1.0 - 1.8 \times 10^{-3}$ in [] corrosion tests. the minimum was 40 mm oil and the maximum about 150 mm oil. 65 cc standard air/sec flowed at $\gamma = 1 \times 10^{-3}$ and $\Delta p = 100$ mm oil. Associating the minimum Δp with maximum γ of 1.8×10^{-3} would give a theoretical air flow through the orifice of $\frac{1.8 \times 40}{1.0 \times 100} \times 65 = 47$ cc standard air/sec

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The maximum Δp associated with the minimum γ of 1×10^{-3} would give a calculated orifice air flow of

$$\frac{150 \times 65}{100} = 97 \text{ cc standard air/sec}$$

These figures were not consistent with 120 cc/sec.

33. [] in general, production diaphragms lay in the range $\gamma = 1.0 - 1.4 \times 10^{-3}$. For corrosion tests, [] chose barriers in this range, but had tested barriers up to $\gamma = 1.8 \times 10^{-3}$. After the corrosion tests, γ had fallen to $0.7 - 1.5 \times 10^{-3}$ due to plugging. The corrosion apparatus itself therefore had to cover a range $\gamma = 0.7 - 1.8 \times 10^{-3}$. [] used several testers, and did indeed determine the constant for each by metering the flow through the ultrapore. [] this constant lay in the limits 100-200.¹⁹

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19. [] Note:

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This statement could be compared with the calculated expression in the theory on pages 16-17.

$$\text{In this } \gamma 10^{-3} = \frac{V \times 760 \times \sqrt{P_1 \times 3 \times 10^3} \times 13.5}{A \times 2 \times 1.59 \times 0.85 \times \Delta p_{\text{oil}}}$$

$$= \text{constant} \times \frac{V}{A \times \Delta p_{\text{oil}}}$$

For fixed $A \approx 250 \text{ cm}^2$, as was almost invariably the case in the tube test:-

$$\gamma 10^{-3} = \text{constant} \times \frac{V}{\Delta p_{\text{oil}}} = \frac{K}{\Delta p_{\text{oil}}}$$

A constant K of 190 was calculated to be associated with $V = 63$ to 126 cc standard air/sec.

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34. [redacted] limits of the
apparatus constant and the limits of Δp . [redacted] 50X1-HUM
limits of γ in terms of 10^{-3} . These were:
smallest possible $\frac{100}{150} = 0.66$
largest possible $\frac{200}{40} = 5.0$
most likely upper $\frac{100}{40} = 2.5$
most likely lower $\frac{200}{150} = 1.33$

It was agreed that $\gamma = 5 \times 10^{-3}$ was out of the question and that the "most likely" range was more realistic, although γ was not likely to exceed 2.0×10^{-3} .

35. Thus one might expect the following associations:

Apparatus constant 100

(Air flow = 63 cc standard air/sec fixed)

$$\left\{ \begin{array}{l} \Delta p \approx 40 \text{ mm oil minimum seen} \\ \gamma \approx 2.5 \times 10^{-3} \text{ maximum seen} \end{array} \right.$$

$$\left\{ \begin{array}{l} \Delta p \approx 100 \text{ mm oil most frequent} \\ \gamma \approx 1 \times 10^{-3} \text{ most frequent} \end{array} \right.$$

Apparatus constant 200

(Air flow = 126 cc standard air/sec fixed)

$$\left\{ \begin{array}{l} \Delta p \approx 150 \text{ mm oil maximum seen} \\ \gamma \approx 1.33 \times 10^{-3} \text{ most likely lower} \end{array} \right.$$

Doubtful $\left\{ \begin{array}{l} \Delta p \approx 100 \text{ mm oil most frequent} \\ \gamma \approx 2 \times 10^{-3} \text{ most frequent} \end{array} \right.$

The above state of affairs was consistent with the use of apparatus with constants of nearer 100 for production testing of barrier in the region of $\gamma = 1 \times 10^{-3}$, when Δp would be nearer figure of 100 mm oil. [redacted]

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36. On the other hand, apparatus with constants of nearer 200 would be more consistent with the corrosion test, which had a known air flow of 120 cc/sec and tested barriers of higher γ (up to 2×10^{-3}).^{50X1-HUM}
37. most likely figure of 120 cc/sec, corresponding to a constant of 190 fixed, although he also thought 100-140 cc/sec had been used. Barriers of the order of $1 - 1.4 \times 10^{-3}$ must then have produced Δp of the order of ~~120-190~~ mm oil.

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QUANTITATIVE SUMMARY OF INTELLIGENCE RELATING TO PORE SIZE

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Barrier Weight²⁰

Mesh Weight

38. With the acid etch of a *diaphragm*, there was a 20 percent loss of weight. With rolling, there ~~was~~ an increase in length of 0.5 cm for a 50 cm long section, with a concomitant loss in thickness and a change in the usable volume of the holes in the mesh.
39. The sheet of wire mesh was 250 cm² in area, allowing enough for one seam, and was 0.1 mm thick. There were 200 cm wire, if taken as straight, in one square centimeter of mesh. The weight was therefore:

$$\frac{d^2 \pi}{4} \cdot 200 \cdot \rho \cdot A = (0.005)^2 \frac{\pi}{4} \cdot (200) (8.9) (250)$$

$$= 8.75 \text{ grams}$$

-
20. This analysis of the weight of a diaphragm had been carried out previously

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49. In fact, however, the waviness of the wire as woven gave a factor of 1.12 on the figure for assumed straight wire. The weight of untreated mesh in the sheet was therefore $1.12 \times 8.75 = 9.8$ grams. For loss in weight of 20 percent by etching, this became $9.8 \times 0.8 = 7.85$ grams.

Sinter Weight

41. The volume of solid nickel in the mesh was $\frac{7.85}{8.9} = 0.88$ cc. The thickness of the mesh was estimated as 0.085 mm, taking into account the loss caused by rolling and etching, so that the over-all volume was $250 \times 0.0085 = 2.11$ cc. The usable volume for sinter was, therefore, $2.11 - 0.88 = 1.23$ cc.

the density of sinter material was about half that of solid nickel, viz., 4.45 gm/cc. The weight of sinter was, therefore, $4.45 \times 1.23 = 5.5$ grams for one sheet of 250 cm area.

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42. The total weight of mesh plus sinter was $5.5 + 7.85 = 13.35$ grams. This figure compared favorably with the 13 grams

The weight of the sheet would have been $250 \times 0.0085 \times 8.9 = 18.9$ grams, if it had been solid nickel. The voidage ratio was, therefore,

$$1 - \frac{13.35}{18.9} = 0.30.$$

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DIRECT DETERMINATION OF PORE SIZE BY BUBBLE METHOD

43. The diaphragm tube was plugged at one end, and the other end was connected to an air supply and manometer. Starting with a considerable over-pressure (more than one atmosphere), the tube was inserted into a vessel containing ethyl alcohol, whereupon numerous bubbles streamed from the surface of the alcohol. The pressure was then reduced until the last bubble stayed steady on the surface, and the pressure difference Δp across the diaphragm was read. Δp , as measured, lay between 300 and 760 mm.

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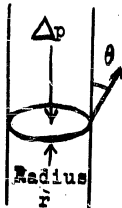
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44.

the test was to determine the radius of the largest pore, and that it was applied to barriers that had failed the three percent Δp test, due possibly to welding faults at the seam.

45.



Assuming that the wetting angle was small, and resolving axially, $\cos \theta = 1$ and $\Delta p \cdot \pi r^2 = \sigma \cdot 2\pi r$, where σ was the surface tension. Therefore, $r = \frac{2\sigma}{\Delta p}$. σ was 25 dynes/cm. Δp was between 0.4 and 1.0×10^6 dynes/cm².

Therefore, the measured size of the maximum pore in a barrier of $\Delta p >$ three percent was in the range $\frac{50}{10^6}$ to $\frac{50}{0.4 \times 10^6}$ cm, i.e., 0.5 to 1.2μ .

46.

the above picture was as simple as possible. In practice, there was a definite value of θ , and the pressures for streaming through the last pore and for a steady bubble just covering the last pore were not the same.

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Determination of Pore Size by Calculation of Barrier Tester Flow

48.

D'Ans and Lax, Taschenbuch fuer Chemiker und Physiker. At the bottom of page 1499 was given an expression for pure Knudsen flow through a straight circular channel of radius r and length l . The mean velocity of flow through such a channel was

$$\bar{w} = \frac{1}{\sqrt{p_1}} \cdot \frac{4}{3} \cdot \frac{r}{l} \sqrt{\frac{8}{\pi}} \cdot \frac{p_1 - p_2}{p_1 + p_2}$$

21.

the figure 0.05 to 0.12 m

is clearly wrong, if his reasoning is to be believed. It is probably another arithmetical errors, and so has been corrected above.

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where

 \bar{w} = mean velocity of flow, cm/sec. ρ = density which the flowing gas would have at one dyne/cm². r = pore radius in centimeters. ℓ = pore length in centimeters. p_1 = upstream pressure in millimeters of oil (or Hg). p_2 = downstream pressure in millimeters of oil (or Hg). $p_1 - p_2 = \Delta p$ measured in millimeters of oil (or Hg).

[Pressure appeared as a ratio, so that any consistent units could be used.]

19. The basic assumption was to take each square centimeter of barrier as a number N of identical pores, each of radius r and length ℓ , passing gas at velocity \bar{w} . 50X1-HUM this model might be far from actuality. The volume flow in cc/sec of actual gas at $(p_1 + p_2) / 2$ mm Hg and standard temperature was

$N \cdot a \cdot \bar{w}$ cc/sec per square centimeter of barrier

= $N a \bar{w} A$ cc/sec for a barrier of area A cm²

where a = cross-sectional area of one pore

$$= \pi r^2 \text{ cm}^2.$$

The volume flow was, therefore:

$$\begin{aligned} V &= N a A \cdot \bar{w} = N a A \cdot \frac{1}{\sqrt{\rho}} \cdot \frac{4}{3} \cdot \frac{r}{\ell} \cdot \sqrt{\frac{8}{\pi}} \cdot \frac{p_1 - p_2}{p_1 + p_2} \\ &= N a A \cdot \frac{1}{\sqrt{\rho}} \cdot \frac{2}{3} \cdot \frac{r}{\ell} \cdot \sqrt{\frac{8}{\pi}} \cdot \frac{\Delta p}{p_m} \end{aligned}$$

$$\text{where } p_m = \frac{p_1 + p_2}{2}$$

and the volume flow was measured in cc/sec at p_m and standard temperature.

50.

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the volume flow was 120 cc/sec of air, referred to 760 mm and 20° C, through a barrier of area

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$A = 250 \text{ cm}^2$, when the upstream pressure was $p_1 = 15 \text{ mm Hg}$ and the pressure difference Δp was $100 \text{ mm oil} = 6.5 \text{ mm Hg}$. Hence,

$\frac{p_1 + p_2}{2}$ was 11.75 mm Hg . Further discussion yielded an

estimated figure of 0.01 cm for l .

11. As an approximation, a value of 50 percent for void-50X1-HUM
age,

 With 50 per-
cent voidage (i.e., 100 percent sinter) and the assumed model,

$$NaA = 0.50 \times 250 = 125 \text{ cm}^2.$$

Finally, the density of air was taken as $1.2 \times 10^{-3} \text{ gm/cc}$ at 760 mm and 20° C . At one dyne/cm², this density would be much smaller, i.e., $1.2 \times 10^{-3} \times 10^{-6} = 1.2 \times 10^{-9} \text{ gm/cc}$, and $\sqrt{p_1}$ would be $3.5 \times 10^{-5} \text{ sec. cm}^{-1}$. The following substitutions were, therefore, made:

$$v = \frac{120 \times 760}{11.75} \text{ cc/sec at } 11.75 \text{ mm Hg and } 20^\circ \text{ C}.$$

$$NaA = 125 \text{ cm}^2.$$

$$\sqrt{p_1} = 3.5 \times 10^{-5} \text{ sec. cm}^{-1}.$$

$$l = 0.01 \text{ cm}.$$

$$\Delta p = 6.5 \text{ mm Hg}.$$

$$p_m = 11.75 \text{ mm Hg}.$$

$$\begin{aligned} \therefore r &= \frac{120 \times 760 \times 3.5 \times 10^{-5} \times 3 \times 0.01 \times 11.75}{11.75 \times 125 \times 2 \times 1.59 \times 6.5} \\ &= 3.7 \times 10^{-5} \text{ cm} \\ &= 0.37 \mu \end{aligned}$$

This was the value of pore radius on the assumed model for assumed pure Knudsen flow at $p_1 = 15 \text{ mm Hg}$. [Any other voidage ratio could be substituted for 0.50 in the denominator.]

Determination of Maximum Permitted Pore Size for Pure Knudsen Flow

52. By direct substitution in the appropriate equation on page 1499 of D'Ans and Lax,

$$\frac{d}{l} < 0.4$$

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where

d = pore diameter

ℓ = mean free path of gas at the operating pressure in the pore
= 3.75×10^{-4} cm at 12 mm Hg.

$\therefore d < 0.4 \times 3.75 \times 10^{-4}$ cm.
 $< 1.5 \times 10^{-4}$ cm.

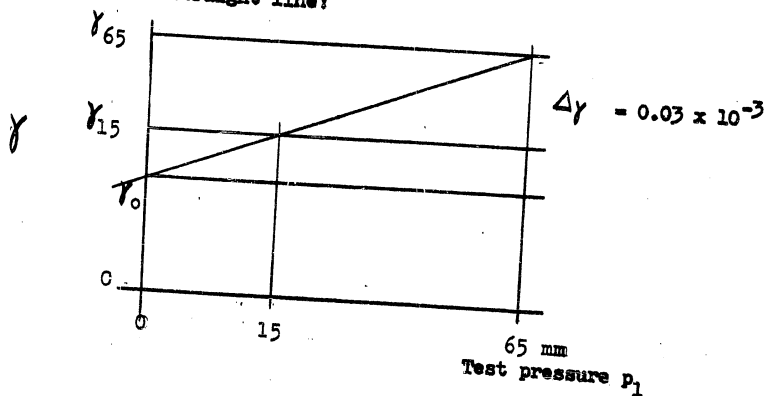
$\therefore d < 0.75 \mu$.

Determination of Pore Size for a Sinter

53. It was possible to determine the pore size for a sinter made up of a number of spheres in contact. [redacted] 50X1-HUM
the "grain size" figure obtained by the Petchukas and Gage method was quite invalid, and was only used as a comparative figure between samples. In actual fact, the carbonyl nickel grains were pencil shaped. It was, therefore, agreed that calculations by this method were unproductive. [redacted] 50X1-HUM

Determination of Pore Size From Poiseuille Component at Upper Pressure

54. [redacted] validity of the reasoning for the third method above, i.e., the determination of pore size by calculation of barrier tester flow, because the flow was about 99 percent Knudsen at $p_1 = 15$ mm Hg. The actual graph of γ with pressure appeared as a straight line: 50X1-HUM



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If γ was calculated on the assumption that the Knudsen theory applied, then the intercept on the γ axis would be γ_0 , corresponding to the true Knudsen component of the flow.

55. In actual fact, γ_{15} came out at about 1×10^{-3} , and the ratio $\frac{\Delta\gamma}{\gamma}$ at about 0.01 to 0.03. [Assuming a straight line, the same $\Delta\gamma$ would be obtained for any pair of test pressures differing by 50 mm Hg.] By proportion, for $\gamma_{15} = 1 \times 10^{-3}$ and $\frac{\Delta\gamma}{\gamma} = 0.03$, $\gamma_0 = (1 - 0.009)10^{-3} = 0.99 \times 10^{-3}$.

56. expression for the Poiseuille component 50X1-HUM of the flow, when the barrier was under $p_1 = 65$ mm Hg. In view of the many inaccuracies already present, it was possible to write that the maximum Poiseuille component at $p_1 = 65$ mm was 0.03×120 cm³/sec of air at 760 mm and 20° C. This was permitted because (a) the pressure difference ($p_1 - p_2$) was maintained sensibly equal to 6.5 mm Hg for both the 15 mm and the 65 mm tests; (b) although the actual volume flow at the test pressure varied, the volume flow was constant when referred to 760 mm and 20° C, as the orifice maintained constant flow; and (c) for small changes, the relative variation of pressure difference at constant flow could be taken as equal to the relative variation of flow at constant pressure difference, assuming the insertion of a parallel Poiseuille flow.

57. The maximum Poiseuille flow in parallel with the Knudsen flow was, therefore, $0.03 \times 120 = 3.6$ cc/sec referred to 760 mm and 20° C. The test conditions were:

$$\Delta p = 6.5 \text{ mm Hg.}$$

$$p_1 = 65 \text{ mm Hg.}$$

$$\text{At } \frac{p_1 + p_2}{2} = 62 \text{ mm Hg, the actual volume flow would be}$$

$$\frac{3.6 \times 760}{62} = 44.2 \text{ cc/sec.}$$

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38. For $N\pi r^2 A$ cm² of pores, each of radius r , the mean Poiseuille velocity \bar{w}_p was, therefore, equal to

$$\begin{aligned} & \frac{44.2}{N\pi r^2 A} \text{ cm/sec} \\ &= \frac{44.2}{125} \\ &= 0.353 \text{ cm/sec,} \end{aligned}$$

assuming $N\pi r^2 A = 125$ cm² as before, i.e., 50 percent voidage and N identical pores, of radius r , per square centimeter.

39. From page 1497 of D'Ans and Lax,

$$\bar{w}_p = \frac{(p_1 - p_2)}{8\eta l} \cdot r^2$$

$$(p_1 - p_2) = 6.5 \text{ mm Hg}$$

$$= \frac{6.5 \times 10^6}{760} \text{ dynes/cm}^2$$

$$= 8.56 \times 10^3 \text{ dynes/cm}^2.$$

$$l = 0.01 \text{ cm.}$$

$$\eta = 1.796 \times 10^{-4} \text{ dyne - cm units.}$$

$$\text{Hence, } r^2 = \frac{0.353 \times 8 \times 0.01 \times 1.796 \times 10^{-4}}{8.56 \times 10^3}$$

$$= 5.92 \times 10^{-10} \text{ cm}^2.$$

$$\therefore r = 2.44 \times 10^{-5} \text{ cm} = 0.24 \mu.$$

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since the velocity of flow varied as r^2 , then the corresponding radius would be $\frac{0.24}{\sqrt{3}} = 0.14$.

Thus in specification barriers, the mean pore radius for Poiseuille flow, based on the same model of N equal cylindrical holes per square centimeter, lay between 0.14 and 0.24 μ .

Collected Results

61. Bubble method: Largest pore for $\frac{\Delta V}{V} > 3$ percent. $0.5 < r < 1.2 \mu$.

Assumed 100 percent Knudsen flow: Mean pore of barrier which passes 120 cc standard air/sec through 250 cm^2 at 6.5 mm Δp and $p_1 = 15$ mm $r = 0.37 \mu$.

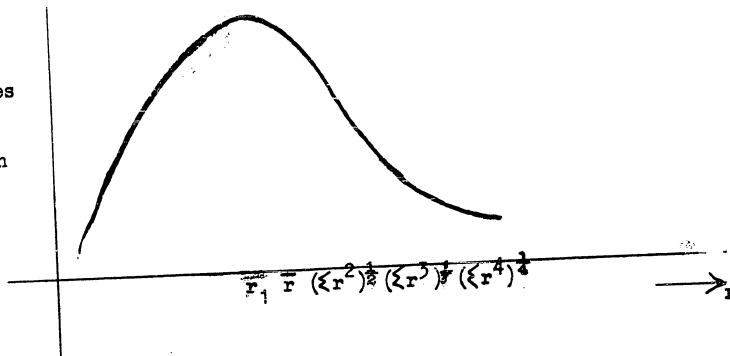
Condition for Knudsen flow: Maximum permissible pore size $r = 0.75 \mu$.

Poiseuille Component in $\frac{\Delta V}{V}$ test: Pore size for assumed Poiseuille flow in range 0.01 to 0.03 of Knudsen flow at $p_1 = 65$ mm, $\Delta p = 6.5$ mm, total flow 120 cc standard air/sec through 250 cm^2 barrier, $0.14 < r < 0.24 \mu$.

Pore Size Variation

62. Some discussion followed on the variation of pore size in a given barrier.

Number
of pores
having
radius
between
 r and
 $r + dr$



The most prevalent radius was r_1 . Outside this value were the mean, root mean square, root mean cube, etc.

the Poiseuille radius ought to lie outside the Knudsen radius, but the exercise was nevertheless considered profitable.

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